PREPARATION OF DENSE ALUMINA CERAMIC BY SLIP CASTING METHOD

A Thesis Submitted

In partial fulfillment of the requirement

For the degree of

BACHELOR OF TECHNOLOGY

Submitted by:

RAHUL ANAND

ROLL NO-110CR0541

SUPERVISOR:

PROF. DEBASISH SARKAR

DEPARTMENT OF CERAMIC ENGINEERING,

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA, ROURKELA-769008

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CERTIFICATE

This is to certify that the project entitled "**Preparation of Dense Alumina Ceramic by Slip-Casting Method**" submitted by **Rahul Anand (Roll No. 110CR0541)** is a genuine work performed by him under my guidance required for the **Bachelor of Technology** degree in **Ceramic Engineering** at National Institute of Technology, Rourkela.

To the best of my knowledge, this thesis is very authentic and none of its matter has been submitted anywhere else for the award of degree or diploma.

Date: 05/05/2014

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Rahul Anand

110CR0541

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ABSTRACT:

Present work focuses on the densification of alumina body by slip casting method. The optimization of slurry and sintering has been done at 1650^{o}C. The alpha-alumina phase of ~1 micron particle size has been used as starting material. Along with alumina some additives like PMAA, MgO-salt, and PVA has been used to prepare slurry. The study of the densification of alumina body at varying soaking time of 2, 4, and 6 hours has also been carried. For better densification of alumina body, MgO salt has been used followed by acid treatment of the slurry. The microstructure of the sintered body is analyzed and densification due to a grain growth has been observed. We are successful to minimize the porosity up to 0.7% and preparing a dense product. The Brazilian disk test and compressive strength testing has been carried out for testing of mechanical property and was found to have good tensile and compressive strength.

CONTENTS

CHAPTER 1

INTRODUCTION

INTRODUCTION

Alumina (Al_2O_3) is one of the most important ceramic oxides which is widely used for structural, electronics, computer, medical and electrical fields, because of the high hardness, good wear resistance, excellent dielectric properties and outstanding mechanical properties. But it has low flexural strength and fracture toughness, i.e. high brittleness, which limits some applications such as for the cutting tools, femoral ball etc. and hence, researchers focus is on the development of material for enhanced mechanical properties [1].

The Na_2O content of alumina is a restricting factor for its industrial use. It must be <0.1% for many applications, including spark plugs and high-voltage insulators, where insulating properties are important. Na_2O content up to 0.5% is allowed for applications where mechanical properties are important [2].

. Alumina commonly occurs in its crystalline polymorphic phase α-Al_2O_3 and is the most thermodynamically stable phase of alumina. Each unit cell of α-Al_2O_3 has rhombohedral plane angle ~55° and having side length 5.2Å. It occurs in numerous metastable crystalline structures also e.g. δ-, θ-, β-, η-, γ-, κ-, χ, and, all of which ultimately form the most thermodynamically stable phase rhombohedral alpha alumina at elevated temperatures [3]. This phase is deliberated as best for structural applications. The main reason for its good mechanical and thermal properties is the close packing of the aluminum and oxygen atoms within its structure. In general there are various shaping methods to prepare ceramic specimens.

Slip casting is normally used for forming of traditional ceramic because it could fabricate green body specimen in complicated shapes with relatively high density and low cost. In slip casting method segregation of particles occur usually in consolidation processes which involve

water removal. In this casting method, binder provides plasticity to the feed material and aid the forming process. There are lots of binders, some of which are soluble in water, and some are soluble in organic liquids. Binders are used for providing strength to the green ceramic body forming bridges between the particles and provide easy handling of the green product. It also affects the density of final sintered product. Amount of binder in slurry affects the rheological properties which may lead to formation of inhomogeneous and flocculated slurry resulting in reduction in density and mechanical strength also. High amount of binders may also lead to formation of porous material. So it is necessary to control the binder amount in slurry. For preparation of green body with high density, major issue is to control slurry viscosity along with uniform dispersion of solid content. For better sintering of ceramic specimen high temperature is required with an appropriate soaking time. Bigot's curves shows very simple and powerful tool to characterize the structure of slip casted bodies. It also enables good correlations between the green microstructure, the rheology of the suspensions and the particle interaction forces.

Grain-size distribution is one of the most crucial parameters in the successful sintering of alumina. Packing characteristic is another critical parameter for sintering. The particle shapes can be needlelike, spherical, tabular, platelet or equiaxial. The best packing of alumina powders is obtained using spherical shapes of various sizes randomly distributed in the batch. The advanced physical and chemical property of alumina ceramic demands ultrafine and high purity alumina powder [4]. By controlling the particle size, shapes and distribution, it is possible to achieve an enhanced densification of specimen with reduced defects in the ceramic product.

MgO can improve sintering of alumina, and in general increases sintering rate and grain growth of ultrapure alumina. MgO increase the densification rate directly by raising the value of grain boundary diffusion coefficient [5, 6]. MgO raises the pore mobility (M_p) on increasing the

surface diffusion [7] whereas it reduces the grain growth rate during sintering by decreasing the surface diffusion [5, 8]. MgO lowers grain boundary mobility M_{gb} (i.e. the mobility of the pore-free segment of the boundary) thus enabling the pore to stay attached to the moving grain boundary during sintering [9].

Particle size influences many properties of suspensions, products and is a good indicator of quality and performance of final product. The size and shape of powders influences flow and compaction properties. Coarser spherical particles flow more easily than smaller or high aspect ratio particles. Finer particles dissolve rapidly and result in higher suspension viscosities than larger particles. Smaller droplet sizes and higher surface charge (zeta potential) improves suspension stability. Finer particles are easy to sinter, therefore can be sintered at lower temperature providing better densification of final product. Reasonably, it is important to measure and control the particle size distribution of the products.

CHAPTER 2

LITERATURE REVIEW

Alumina (Al_2O_3) has wide application as engineering ceramic material due to its high hardness value. It has high melting point (2054 °C), low thermal expansion and high compressive strength which provides good thermal shock resistance [10]. Alumina has good electrical insulation at high temperatures, good wear resistance and high hardness, makes it suitable for components such as ball valves, piston pumps and deep drawing tools. For machining and grind of alumina diamond tools are needed.

Alumina forms solid solutions with some oxides and low melting eutectics with silica and several other oxides. The Al^{3+} and O^{2+} ions have relatively high mobility at high temperatures, and hence it can be sintered easily. Alumina can be used in pure form and also in alloying component in aluminum oxide based ceramics which contain greater than 85% Al_2O_3.

Various methods has been used to preparation of alumina powders depending on the desired particle size and purity, for example thermal decomposition of aluminium containing salts, dehydration of aluminum hydroxide, mechanical grinding of fused alumina. Some properties of AI_2O_3 are listed in Table 2.1. [10]

Properties	**Unit**	**Alumina**
Density	g/cc	3.98
Tensile strength	MPa	300-900
Hardness	Hv	2200
Young modulus	GPa	380
Fracture toughness	$MPa.m^{1/2}$	4.40

Table 2.1 Mechanical properties of Al_2O_3 [11]

Commonly, the green density effects the sintering of the product. The green density can be controlled with pressure in powder pressing or dispersion conditions in slip casting as observed by **Carolina Tallonet al [12].**

Hotta et al [13] observed that small amount of coarse particles governed the fracture strength of sintered body. The strength variation of the sintered bodies is related with the size distribution of coarse particles.

Hotta et al [14] in another research observed that the strength of the body decreases continuously with the coarser particles. No fracture origin other than the coarse particles has been found in study. The fracture strength σ is related to the size of fracture origin c as follows,

$$\sigma = K_{IC}/Yc^{1/2}$$

Where K_{IC} is fracture toughness and Y is the shape factor [15].

Tari et al [16] has observed that the stabilizing mechanism affects the effective solids volume fraction of the suspended particles and so, the rheology of the suspensions and the packing behaviour during the slip casting process is also affected. At low solid loading high zeta potentials promoted the formation of more regular and ordered, when the solid loading increased, the green density obtained from electro- statically stabilized systems using a dispersant, tended to increase, owing to a reduction of the segregation phenomena, until a maximum was reached. This was followed by a slight decrease because of mutual interferences of particles during deposition. The total shrinkage always reduces with increasing solid loading in suspension.

Radonjic et al [17] studied the effect of MgO doping on the densification of alumina. It effects the grain growth of alumina. It was observed that magnesia addition increases the densification

kinetics of alumina but decreases the grain growth rate. The main function of magnesia addition was to decrease the particle size during phase transformation and grain growth during sintering.

Radonjic et al [18] in another research has reported comparative results of alumina and magnesia doped alumina and found that later can be sintered at a low temperature. Magnesia addition decreases the particle size and increases the densification rate. No abnormal grain growth was observed beyond 1200^{o}C. By increasing the MgO addition, the grain size of alumina decreases that means MgO retards the grain growth of alumina.

Ikegami et al [19] reported that addition of MgO decreases the shrinkage rate and the surface tension of Al_2O_3. In addition to that, another literature mentions enhancement in densification by lowering the surface diffusion **[20].**

Olhero et al [21] reported that the suspensions having broader particle size distribution presents a more visible particle size separation on comparison with the ones with a narrower distribution.

Hotta et al [22] studied that grain size and relative density of the sintered compacts produced by slip casting using gypsum molds is influenced by the effect of acid treatment. It was observed that microstructure of sintered compacts without acid washing was found to have discontinuous grain growth. The heterogeneous grains became bigger with rise of sintering temperature. On the other hand, the grains of the sintered compacts with acid was found homogeneous, even at higher sintering temperature also. The relative densities of the sintered compacts with acid wash were higher than that of the sintered compacts without acid-wash. With increasing sintering temperature, the difference of the relative densities of the sintered compacts with and without acid-wash became larger. Not only in the case of high grade Al_2O_3 but also the grains of sintered compacts of the low-soda Al_2O_3 produced by the acid treatment grow homogeneously at higher temperature. Thus, ceramic compacts with a fine grained and uniform microstructure is desirable

for ceramic applications to produce a reliable structural part that can be easily obtained by acid treatment of the slip-casted body.

Nettleship et al [24] observation was based on alumina slurry when slip casted in the dispersed and the flocculated state. A significant difference in the kinetics of densification and effect on the microstructure was found but there was no intrinsic hindrance to the densification of flocculated casts of alumina at low temperature. Even low temperature sintering of flocculated alumina will give high density but the sintering times is very long. From their observation it can be concluded that microstructural pathways based on average microstructural parameters are unaffected to the effects of the casting condition in the absence of distinct grain growth i.e. microstructural pathways for grain clusters is more thoughtful than casting conditions.

OBJECTIVE OF THE WORK:

Thus, aim of the present work is to develop dense alumina ceramic specimen from calcined alumina using slip casting method. This work focuses on enhancement of density as well as mechanical property of alumina ceramic for which solid content and water amount is controlled to make homogeneous slurry to get a dense product.

CHAPTER 3

EXPERIMENTAL WORK

3. EXPERIMENTAL WORK

3.1 XRD ANALYSIS

The presence of alumina phase in the used powder is known from the X-ray diffraction measurement of powder. When X-rays passes through matter, the radiation interacts with the electrons in the atoms, resulting in scattering of the radiation as shown in Figure 3.1. For crystalline materials the distances between the planes is same and if the atoms are of the same magnitude as the wavelength of the X-rays, constructive and destructive interference will occur. This diffraction where X-rays are emitted at characteristic angles based on the spaces between the atoms organized in crystalline structures are called planes.

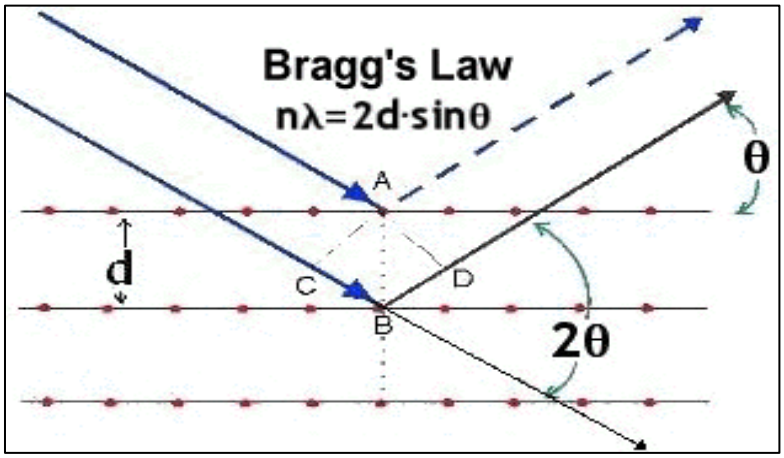

Figure 3.1: Study of crystallographic plane during XRD

3.2 PARTICLE SIZE DISTRIBUTION AND ZETA POTENTIAL

A suspension of alumina powder is prepared with 0.3 gram alumina powder in 50ml distilled water. This suspension is kept for ultrasonication for 5 minutes. The temperature of the SUSPENSION IS maintained below 35°C. Ultrasonic energy is given to the suspension for homogeneous separation of suspended particles. This suspension is injected into a cuvette and

particle size is analyzed. The plots of particle size distribution versus intensity and volume is observed. Similar method is followed for zeta potential analysis in the same set up connected to a computer.

Principle:

Laser diffraction measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles. The angular scattering intensity data is then analyzed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering.

3.3 SLURRY PREPARATION

Slurry is prepared using alumina as raw material with distilled water, concentrated nitric acid, ammonium polymethacrylic acid 0.1% solid loading (Darvan C), polyvinyl alcohol (PVA, 3wt% solution), and magnesium nitrate salt as additives. They are magnetically stirred for homogeneous mixing and milling at 1200 rpm for 24 hours. A flow chart describes the whole slurry preparation method in fig: 3.2 and corresponding to that an image of instrument and equipment used for the slurry preparation is shown in Figure: 3.3.

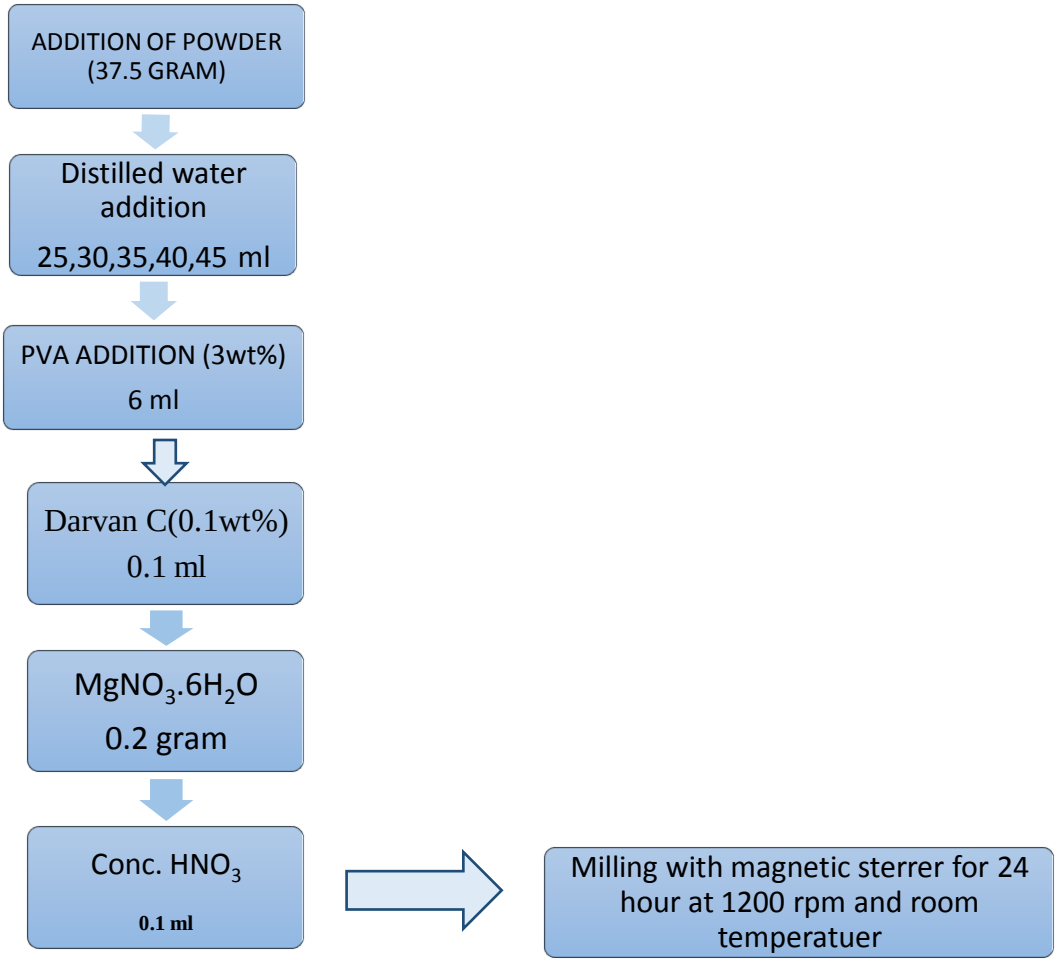

Figure 3.2: Flowchart for preparation of slurry

Figure 3.3: Slurry preparation set-up

3.4 MOLD PREPARATION

A glass plate is taken over which the oil is applied. This oil acts as a lubricant and does not allow the mold to stick on the glass. Then a cardboard is taken using which a hollow cubic shaped body is prepared. The dimension of hollow part should be same having equal and enough distances. This helps in uniform drying during the casting process. Slurry is prepared using water and Plaster of Paris in the ratio 1:1. Wet sand is used to cover the gap between the cardboard and the glass plate to avoid leakage of slurry from the holes or gap between them. Then the prepared slurry is slowly poured in to the hollow cubical box to a certain height. The body is kept for air drying for about 24hrs. After 24hrs the body is removed from the cardboard and kept for air drying for another 24hrs. Then the mold is kept in drier at temperature 60 to 70^{O}C for complete removal of water. The mold inner surface was polished using a sand paper after drying. Figure 3.4 shows the typical designed mold using the above procedure.

Figure 3.4: Top-view of designed mold

3.5 CASTING OF GREEN PRODUCT

The prepared alumina slurry is poured into the designed mold. Since, water of slurry is instantaneously being absorbed by the plaster mold; therefore, continuous slurry is added to fill the hollow. This is repeated till the time when there is no more water absorbance is seen through the mold and hardening of the slurry starts. Solid casting is done. After 2 hour demolding is done. The green product leave the wall of plaster mold and due to shrinkage the product comes out from mold automatically. This green product is of pellet shape. The flowchart of the experimental work has been shown in Figure 3.5.

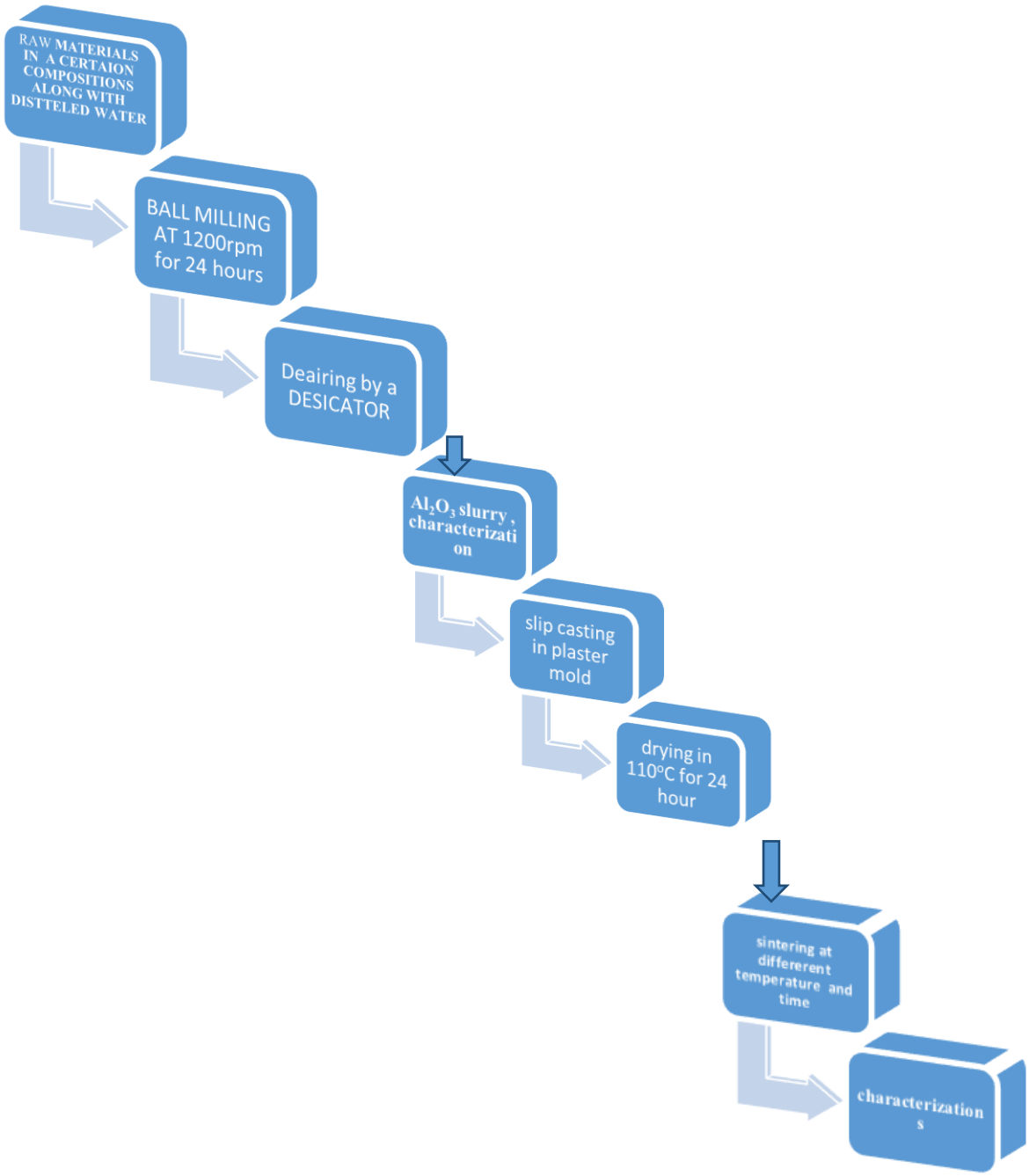

Figure: 3.5 Flowchart of experimental work

3.6 DRYING AND SINTERING

Green pellets are dried under atmospheric condition for a period of around 24hrs at RT. Removal of some water occurs at this stage. Then the green pellets are kept in oven at 110^{o}C for complete

removal of water (~99%) for a period of about 24hrs. Then green pellets are polished by a sand paper to remove contamination of impurities on the surface of alumina pellet. After this sintering of green pellets are done at under different temperatures. The sintering has been done according to the following sintering profile shown in Figure 3.7. The images of sintered pellets are show in Figure 3.8 below.

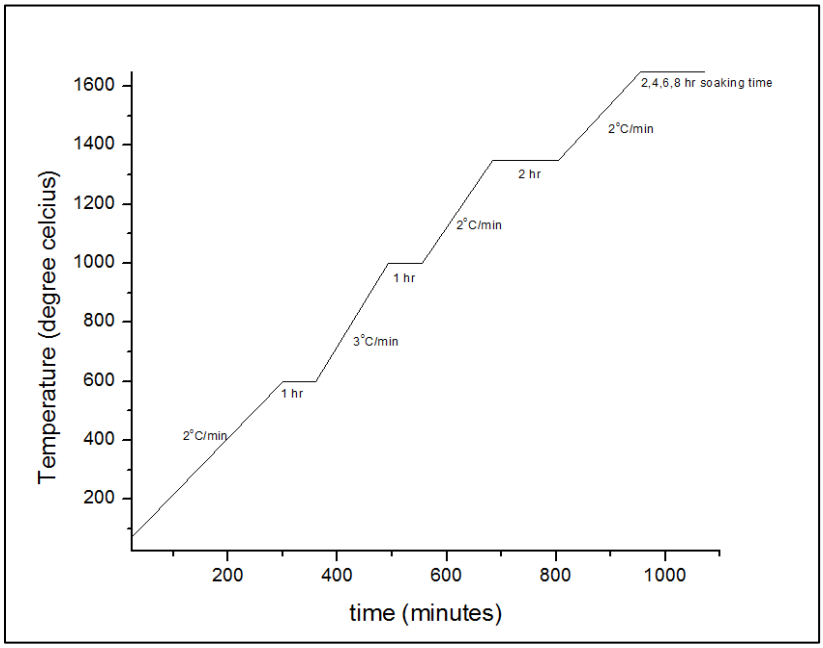

Figure 3.6: Temperature profile during sintering process

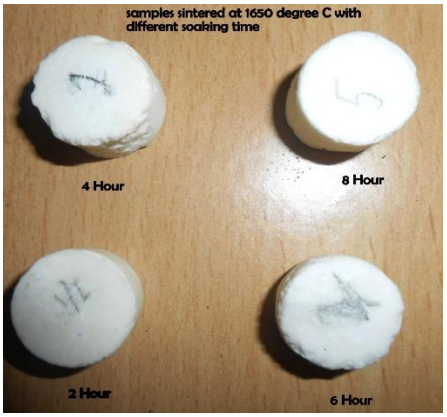

Figure 3.7: Sintered Pellets at different sintering duration

3.7 CHARACTERISATION OF PHYSICAL PROPERTIES OF ALUMINA SPECIMEN

As known, inspection and testing of refractory is important to ensure the use of the end product. Apparent porosity (AP), bulk density (BD) and percent volume shrinkage is determined to check the physical properties. Furthermore, mechanical testing (Brazilian disc test, Hardness and fracture toughness) and microstructure analysis is carried.

3.7.1 DETERMINATION OF AP & BD

Highly porous specimen has low strength, poor heat conducting property & high permeability that is poor resistance towards penetration of slags, corrosive chemicals and gases. BD determines the overall weight coming upon the foundations i.e. strength. A good specimen should have low porosity & high bulk density.

The green sample fired at 1400^{o}C, 1500^{o}C, 1650^{o}C (pellets) are weighed with 0.01 gm accuracy. This is called dry weight (D). The dry specimen is placed in a beaker and is filled with distilled water. This beaker is kept in desiccator for 30 minutes & evacuated to a pressure of less than 25 mm of Hg. The specimen is allowed to remain under reduced pressure for 5-6 hours after which air is allowed to enter the desiccator. Test specimen is suspended with the help of pan in distilled water and suspended weight of sample in water is taken which is called suspended weight (S). Now liquid drops appearing on the surface of sample are wiped & weight is taken in air. This is called soaked weight (W).

Calculations:

The apparent porosity (AP) and bulk density (BD) is calculated as:-

$$\text{AP} = \frac{W-D}{W-S} \times 100\ (\%)$$

$$\text{BD} = \frac{D}{W-S} \times density\ of\ the\ media\ in\ which\ expt.is\ performed$$

Here, distilled water is used as media (density of distilled water is = 1.00 g/cc).

3.8 MICROSTRUCTURAL ANALYSIS

Initially the samples are cut into small pieces; these pieces are rubbed on diamond grinding cloth in order to make its surface plane. These samples are mounted with the help of Bakelite followed by hot pressing at temperature around 135^{o}C for 12 minutes and load is applied of 20 Kilo Newton (KN). Then three consecutive polishing is done to the sample (6µm, 3 µm, and 1 µm cloth) using diamond suspension spray. Ultra cleaning of samples is performed after each polishing. For this purpose samples are immersed into the solution of the distilled water and soap oil. Mounted sample is put on a heater for burn out of Bakelite. Thermal etching is done for each sample in order to get separated grain boundaries so that better microstructure is obtained.

3.9 EVALUATION OF MECHANICAL PROPERTY

3.9.1 COMPRESSIVE STRENGTH

Compressive strength of sintered body is calculated using the Universal Testing Machine. It is a mechanical press used for circular pellets with a range of 10 KN and it measures the maximum force on a pellet and also its compressive strength. The sample is kept towards the center and once the operation starts the force increases until the crack occurs in sample and after that force decreased considerably remaining constant for some time. The maximum force is at the point of failure of specimen. This force is used to calculate the compressive strength of specimen.

Calculation:

$$\text{Compressive Strength} = \frac{F_{max}}{\pi D^2/4}$$

Where, $F_{max}$is the force applied for failure and D is the diameter of the circular pellets.

3.9.2 BRAZILIAN DISK TEST

The mechanical strength of sintered samples is estimated by the diametral compression method called "Brazilian test". Just after sintering, the disk-shaped samples were inserted in the universal testing machine. The load F applied radially at a speed rate of 0.5mm mn^{-1} until the specimen is fractured.

Calculation:

The strength of samples is calculated using the following expression

$$\sigma = 2F_{max}/\pi De$$

Where F_{max} is the load applied at fracture point, and D & e are the diameter and the thickness of the sample, respectively. The Universal Testing Machine for Brazilian test has been shown in Figure 3.10.

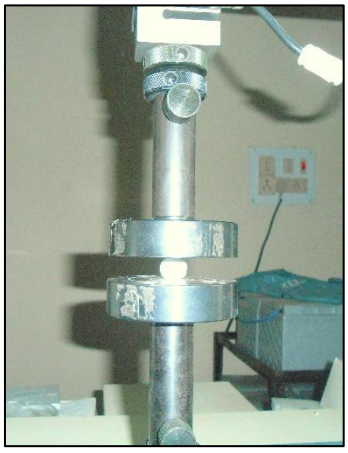

FIGURE: 3.10 Brazilian test by Universal Testing Machine

CHAPTER 4

RESULTS AND DISCUSSION

4.1 XRD ANALYSIS

The room temperature XRD pattern shows that the powder is composed of α-alumina phase as found on comparing with JCPDS file no. 82-1399 (Figure 4.1).

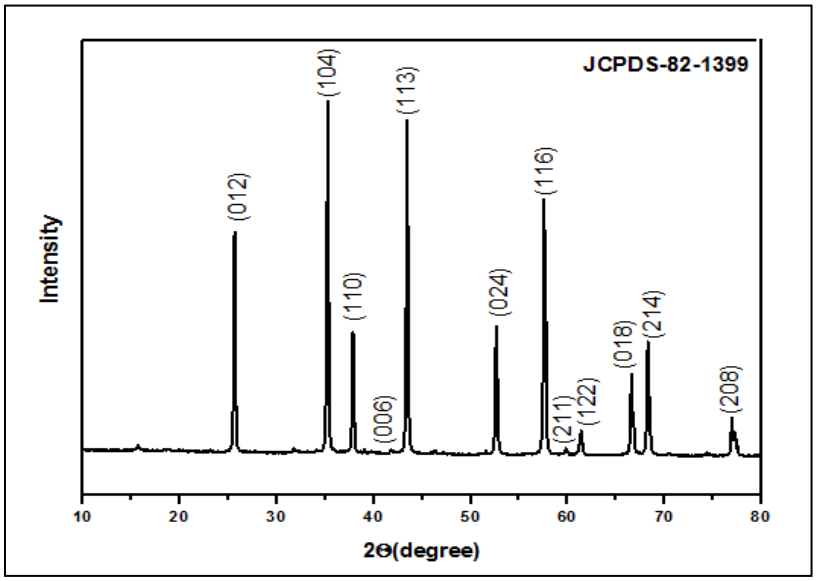

Figure 4.1: XRD phase identification of starting powders

4.2 PARTICLE SIZE DISTRIBUTION

Particle size distribution helps us to understand about the average particle size and range of the particles present. Fig. 4.2(A) shows the particle size distribution of the raw alumina powder. The particle size measurement reveals mono-modal size distribution. Average particle size has been found to be 1.107 micron and the particle size has been found to be in the range of 0.825 micron to 1.718 micron from the figure.

It can be observed that the maximum volume particles (~30.5%) are finer than 1 micron (Table 4.1). The statistical graph for particle size distribution also shows the similar results as shown in Fig.4.2(B).

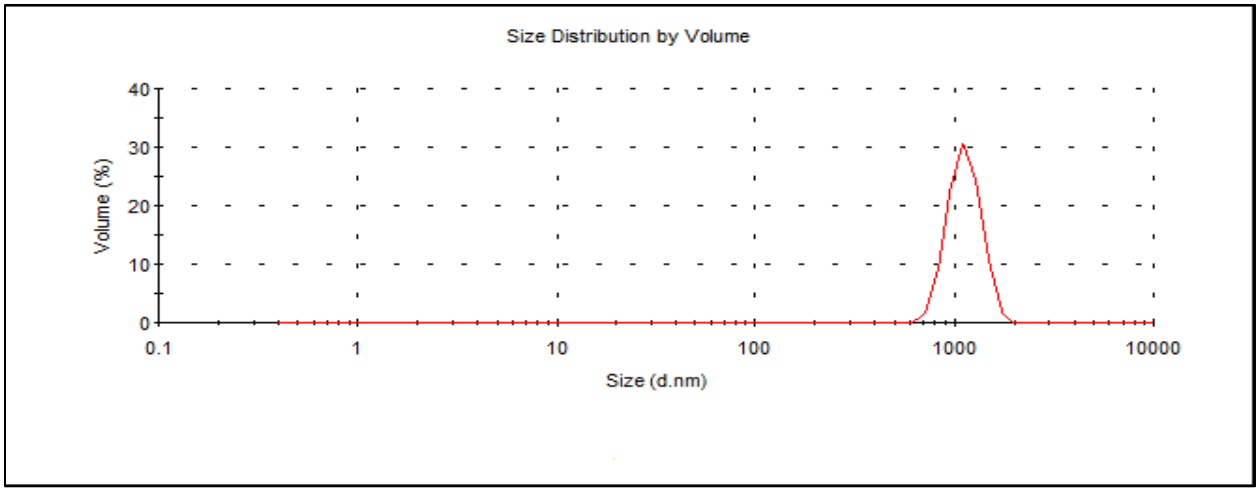

Figure 4.2 (A)

Particle size (nm)	Volume %	Mean Intensity (%)
825	9.3	9.1
955.4	22.6	27.9
1106	30.5	34.9
1281	24	22.9
1484	10.2	5.2
1718	1.8	0

Table: 4.1: Particle size distribution with volume and intensity

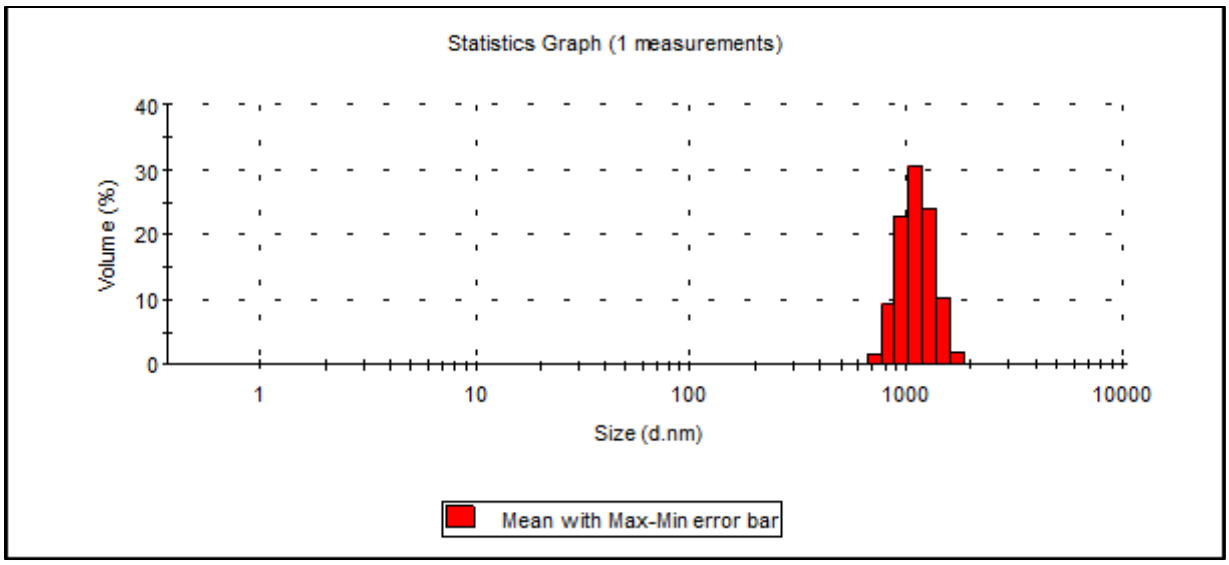

Figure 4.2 (B)

Figure 4.2: Particle size distribution of starting powders.

4.3 ZETA POTENTIAL

Zeta potential of alumina suspension and prepared alumina slurry is found to be -25 mV and +51.8 mV, respectively. To prepare a highly dense product we need a very stable slurry. DarvenC is used as a dispersant here that prevents flocculation and provides a better homogeneity to the slurry. A homogeneous slurry provide better strength to the sintered product as compared to flocculated slurry. Figure 4.3(A) and Figure 4.3(B) shows the zeta potential values of alumina powder suspension and the prepared alumina slurry. So from the zeta potential value it is obvious that the alumina slurry prepared is highly stable.

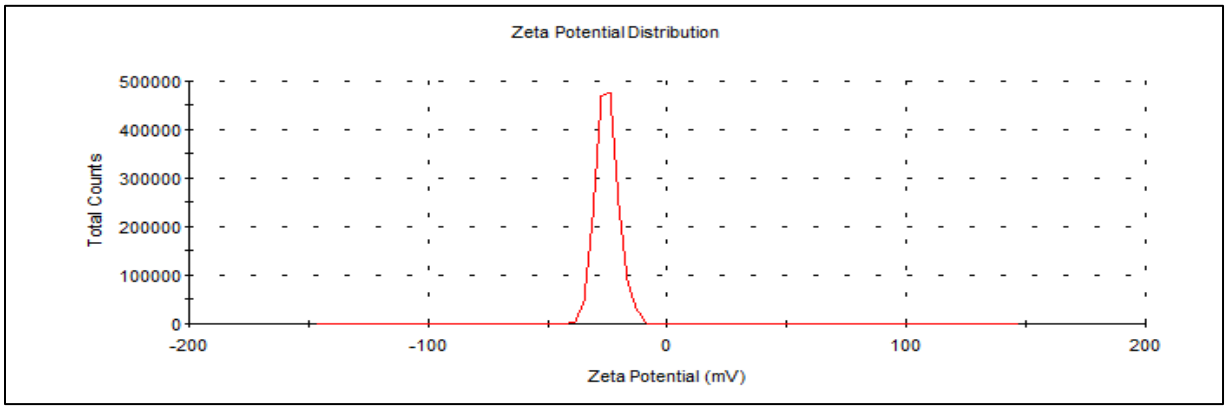

Figure 4.3 (A)

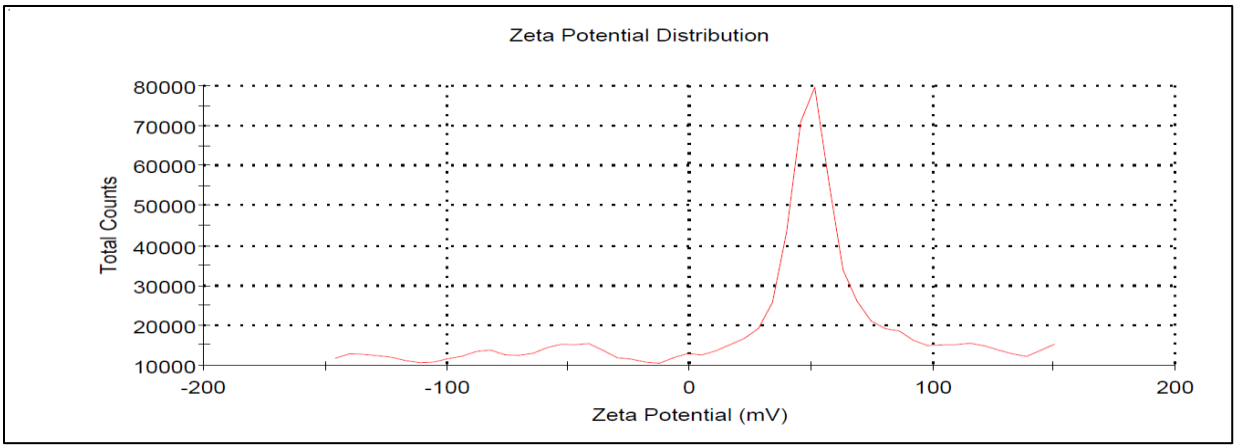

Figure 4.3 (B)

Figure 4.3: The zeta potential of alumina suspension (A) and slurry (B)

4.4 APPARENT POROSITY AND BULK DENSITY

From Table 4.2 we can observe that as solid content of a homogeneous slurry increases the density is also increased however it also depends upon the flow behavior of the slurry. The less viscous slurry or highly viscous slurry causes the flocculation of alumina particles. As with high alumina loading the particles come closer enough and hinders the movement of each other because of attractive forces. An insignificant repulsive force starts acting among them that leads to agglomeration of dispersed particles. For less viscous slurry (means slurry with high solid content), at an optimal condition particles starts settling because of gravitational forces. Here all the samples are sintered at 1650^{o}C for 2 hours.

Table 4.2 The AP & BD variation with water content of slurry

Water amount(ml)	AP (%)	BD(avg.) (g/cc)
45	8.2	2.850
40	**6.31**	**3.314**
35	6.63	3.110
30	7.79	2.960
25	8.01	2.892

We can observe that AP and BD is dependent on the solid content of slurry. From Fig. 4.4 we can observe the highest density & least porosity have been found for 40 ml water content.

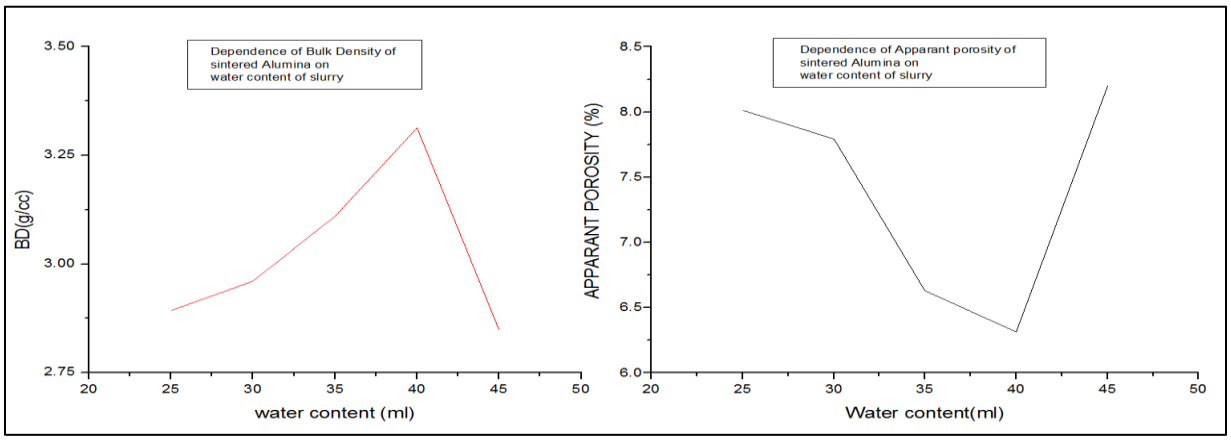

Figure 4.4: Dependence of Bulk Density and Apparent Porosity of sintered Alumina on water content of slurry.

Taking the optimized composition of the slurry containing 40 ml water, the green casted specimens are sintered at 1650°C with 2, 4 and 6 hours to see the variation in density of alumina specimen with respect to soaking time. The soaked samples at 2, 4 and 6 hours soaking is termed as S-1, S-2, and S-3 respectively. Table 4.3 shows the AP and BD of the sintered specimens. It was observed that as the sintering time is increased the density also raises and the apparent porosity minimizes. As the soaking time increases the complete sintering of specimen occurs by liquid sintering. We can see that the maximum density (3.426g/cc) and minimum porosity (0.70%) is for 6 hour soaking and the minimum density (3.314) & maximum porosity (6.31%) is for 2 hour soaking as represented in Fig. 4.5.

Table 4.3 Variation of AP & BD with soaking time whereas the sintering temperature is 1650°C

Sample name	Soaking time (in hrs.)	AP (%)	BD (avg.) (g/cc)
S-1	2	6.31	3.314
S-2	4	1.80	3.362
S-3	6	0.70	3.426

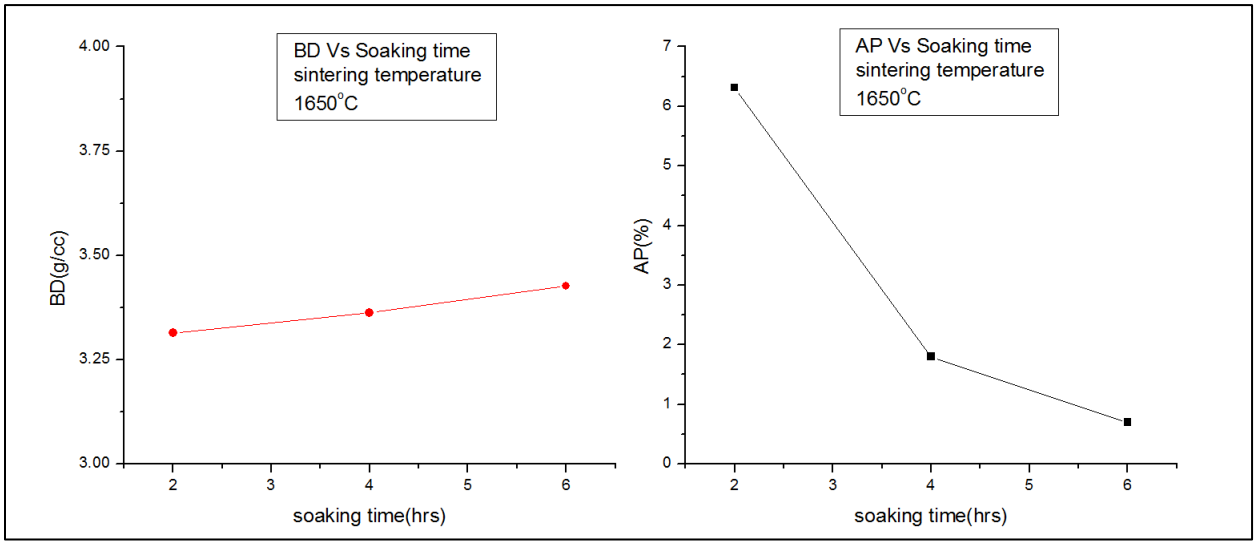

Figure 4.5: Dependence of Bulk Density and apparent porosity to soaking time for sintered alumina at 1650°C.

4.5 MICROSTRUCTURE STUDY

Figure 4.6 shows FESEM micrographs of the alumina product of the particle size ranging 800-1400nm. In the micrograph at higher magnification it can be observed that as the sintering time increases the grain size is also found increasing. Figure 4.6 (A) shows microstructure of the alumina compact sintered at 1650^{o}C for 2 hours. The grain size is in range of 400-700 nm. The Fig. 4.6(B) shows the micrograph of the product sintered at 1650^{o}C for 4 hour. The grain size for this specimen is 2-4 micron and for the product sintered at the same temperature for 6 hours has grain size 3-5 micron. It is found that the grain size increases significantly. Figure 4.6(D) shows the shape of the pore to be of triangular shape. The grain shape in Fig. 4.6 (B) is spherical which doesn't provide the best packing. In The 4.6 (C) elongated grains shape is observed which provides better densification than spherical grains. It is found that the relative density greater than 83.27% can be achieved after the compact was heated at 1650^{o}C for 2hours. For the product of holding duration being 6 hours, 86.08% has been achieved as a result of high compact packing of grains and very small pores are observed which leads the dense alumina formation, and provides good mechanical property.

We can see the MgO particles at grain boundaries in microstructure. The MgO addition and acid treatment of the slurry provided a better densification to the final sintered product. MgO acts as grain growth rate inhibitor and increases the grain size by increasing the grain boundary diffusion constant and lowering the surface diffusion.

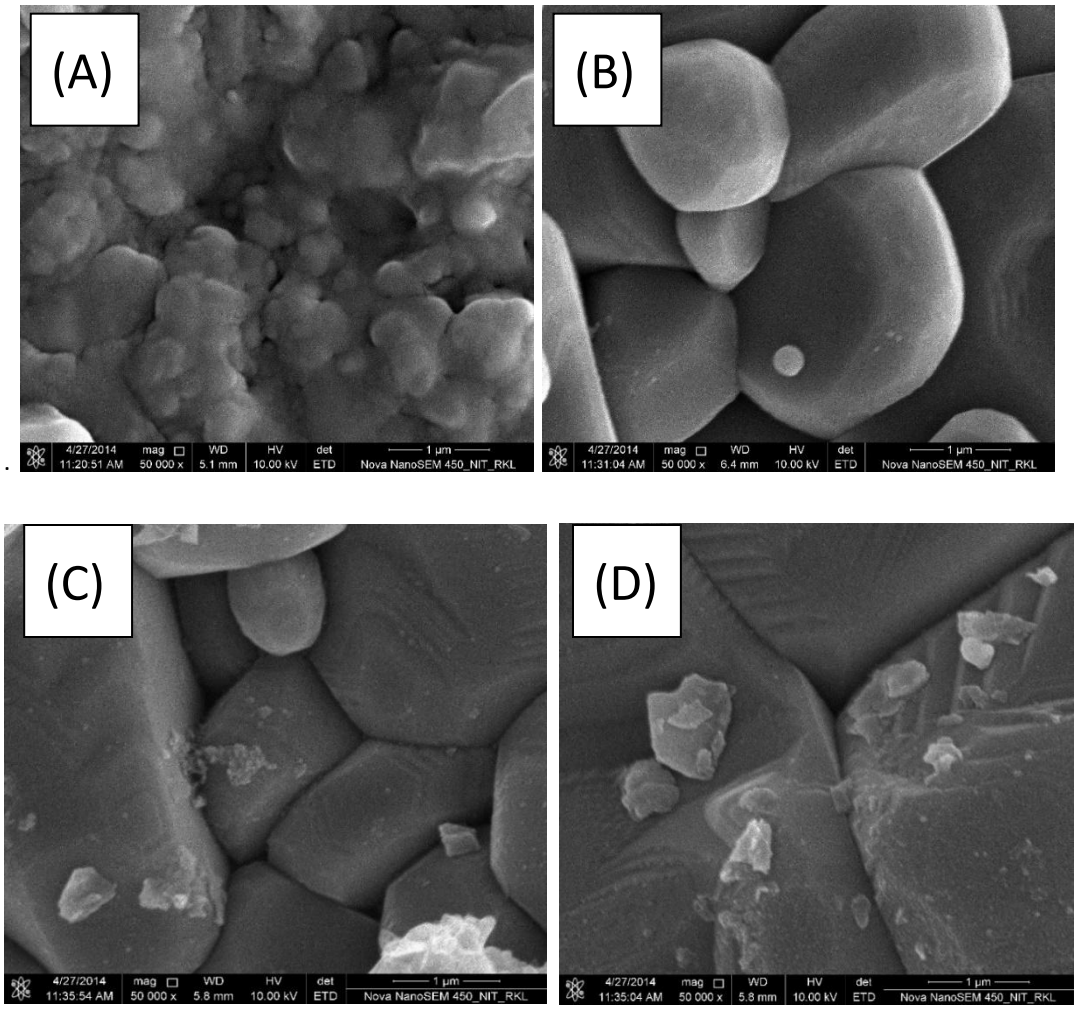

Figure: 4.6: FESEM micrographs of the sintered samples at 1650^{O}C for 2 (A) and 4 (B) and 6 hours (C) and (D).

The main function of magnesia addition is to decrease the particle size during phase transformation and to improve grain growth during sintering. Grains of the sintered compacts with treatment is found to grow homogeneously, even at higher sintering temperature also and provides a better relative density product after sintering than without acid treated slip casting as observed from the results.

4.6 MECHANICAL TESTING

The tensile strength is measured by Brazilian disk test. The comparative study of compressive and tensile strength of the product sintered at 1650^{o}C for different soaking time has been shown in Table 4.4 and 4.5 listed below. As observed, the sample with 1650^{o}C and 2 hour soaking is found to have good compressive & tensile strength (61.96 MPa & 38.504 MPa respectively but we can enhance the mechanical strength by increasing the sintering time of the specimens. The compressive strength and tensile strength both increases with the holding time of compact. The sample with soaking time 6 hours has compressive strength 72.8 MPa and tensile strength 62.65MPa. This shows the sample (S-3) has a good mechanical strength.

Table 4.4: test result of compressive strength of samples with different samples.

Sample name	Compressive Strength (MPa)
S-1	61.96
S-2	67.30
S-3	72.82

Table 4.5: Test result of tensile strength by Brazilian disk method.

Sample name	Diameter(D) (mm)	Thickness(e) (mm)	F_{max} (Kilo N)	Tensile strength(σ) ($\sigma = 2F_{max}/\pi De$) (MPa)
S-1	16.35	8.84	8.745	38.50375
S-2	16.51	4.10	6.027	56.66091
S-3	16.70	4.22	6.938	62.64965

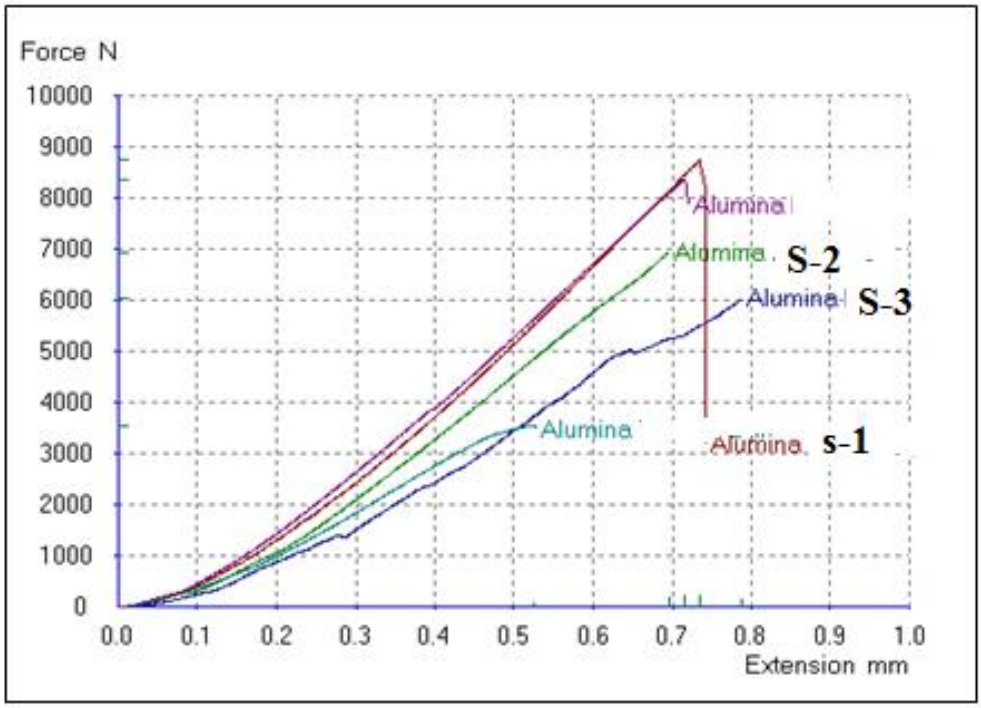

Figure: 4.7: Tensile strength curve

CHAPTER-5

CONCLUSIONS

In the present study, an Al_2O_3 body is made by slip casting method which contains inherent characteristic packing structure of powder particles. The flow character of slurry with water and its effect on density and microstructure has been observed. The excess and low water in slurry leads to the flocculation of particles forming non-homogeneous slurry. The optimum condition for homogeneous slurry is found to be addition of 40 ml water for slurry preparation. It is observed that the relative density of specimen casted from flocculated slurry has less density than the specimen made from the dispersed slurry.

The effect of the soaking time to the density of sintering product is observed. It has been observed that with the increase in holding time the sintering improves i.e. the densification enhances and the porosity minimizes. It is clear from the microstructure that the product of the maximal densification have largest grains. Because of the acid treatment, homogeneous grains in final body developed which helps in better densification. We achieved 86.08 % densification of given Alumina powder. The maximum bulk density is 3.426 found and minimum apparent porosity is 0.7% observed of sample sintering at 1650^oC for 6 hours soaking.

The compressive and tensile strength is measured which clarifies that the product has good compressive and tensile strength. The improved densification is because of its enhanced densification. It is also clear as alumina body becomes denser its mechanical property (compressive and tensile strength) also improves. The compressive and tensile strength is found varying from 61.96 to 72.8 MPa and 38.50 to 62.65 MPa, respectively with 2-6 hour sintering at 1650^oC. Summarizing, the densification of alumina is done by the controlling the particle size, the water content of slurry, by increasing the sintering temperature and soaking time. Acid treatment and MgO addition also helped in densification and a homogeneous growth of grain size forming a dense compact of alumina body.

REFERENCES:

1. Xinying Teng, Hanlian Liu, Chuanzhen Huang, Effect of Al_2O_3 particle size on the mechanical properties of alumina-based ceramics, Materials Science and Engg. 452–453 (2007) 545-551.

2. Turan Tambas, Z. Engin Erkmen and Serdar Ozgen, Improvement on the Slip Casting of Bayer-Processed Alumina, American Ceramic Society Bulletin, Vol. 86, No. 2.

3. Paglia G. (2004). "Determination of the Structure of γ-Alumina using Empirical and First Principles Calculations Combined with Supporting Experiments", Curtin University of Technology, Perth. Retrieved 2009-05-05.

4. V.K. Sing, R.K. Sinha, Mater. Lett. 18 (1994) 201–206.

5. R.J. Cable, J. Appl. Phys., 32 (1961) 793-799.

6. M.P. Harmer and R.J. Brook, J. Mater. Sci., 15 (1980) 3017-3024.

7. A.H.Heuer, Journal of American Ceramic Society. 62 (1979) 312-318.

8. R.J. Brook, Proc. Br. Ceram. Sot, 32 (1982) 7-24.

9. S.J. Bennison and M.P. Harmer, J. Am. Ceram. Sot, 68 (1985) C22-24.

10. http://surphv.fat.bme.hu/pub/MaterSci/Tl0.DQC.pdf 04-07-2005 (ppl71)

11. L. Richard, Lehman Said K. El-Rahaiby john B. Wachhtman, Jr. Handbook on continuous fiber-Reinforced ceramic matrix composites, (1999) pp 119-122.

12. Carolina Tallon, Monika Limacher, George V. Franks, Effect of particle size on the shaping of ceramics by slip casting, Journal of the European Ceramic Society 30 (2010) 2819–2826.

13.Tadashi Hottaa, Hiroya Abeb, Makio Naitob, Minoru Takahashic, Keizo Uematsud, Zenji Kato, Effect of coarse particles on the strength of alumina made by slip casting, Powder Technology 149 (2005) 106– 111.

14. Tadashi Hotta, Hiroya Abe, Makio Naito, Minoru Takahashi, Keizo Uematsu, Zenji Kato, Effect of coarse particles on the strength of alumina made by slip casting, Powder Technology 149 (2005) 106– 111.

15. G.K. Bansal, Effect of flaw shape on strength of ceramics, J. Am. Ceram. Soc. 59 (1976) 87-88.

16. G.Tari,J.M.F.Ferreira,and Lyckfeldt, Influence of the Stabilizing Mechanism and Solid Loading on Slip Casting of Alumina, Journal of the European Ceramic Society 18 (1998) 479-486.

17. Yung-Fu Hsu, Sea-Fue Wang, Yuh-Ruey Wang, Shih-Chueh Chen, Effect of niobium doping on the densification and grain growth in alumina, Ceramics International 34 (2008) 1183–1187.

18. L. RadonjiC, V. SrdiC, Effect of magnesia on the densification behavior and grain growth of nucleated gel alumina, Materials Chemistry and Physics 47 (1997) 78-84.

19. Takayasu Ikegami, Nobuo Iyi, Isao Sakaguchi, Influence of magnesia on sintering stress of alumina, Ceramics International 36 (2010) 1143–1146.

20. R.J. Brook, Proc. Br. Ceram. Soc., 32 (1982) 7-24.

21. S.M. Olhero, J.M.F. Ferreira, Particle segregation phenomena occurring during the slip casting process, Ceramics International 28 (2002) 377–386.

22. Yuji Hotta, Microstructural changes in sintered Al2O3 by acid treatment of compacts produced by slip casting in gypsum molds, Ceramics International 28 (2002) 593–599.

23. Ian Nettleship, Richard McAfee, Microstructural pathways for the densification of slip cast alumina, Materials Science and Engineering, A352 (2003) 287-293.